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THE APPLICATION TO A SOFT-WATER SEWAGE OF DIRECT PROCESSES FOR THE DETERMINA- TION OF KJELDAHL NITROGEN AND NITROGEN AS FREE AMMONIA

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WHEN the Experiment Station at Waterbury, Connecticut, was started in operation, December, 1905, it was decided to adopt direct processes for the determination of Kjeldahl nitrogen and nitrogen as free ammonia.

After reviewing the literature on the direct determination of Kjeldahl nitrogen, it was decided to apply the method described by Kimberly and Roberts,¹ and used at the Columbus Sewage Testing Station. In carrying out this method under Waterbury conditions, it was soon found that several points of moment under Columbus conditions did not affect the applicability of the method for this soft-water sewage, and hence it was possible to simplify the technique. It is the purpose of the writer to outline briefly the experiments at Waterbury with direct nesslerization in Kjeldahl nitrogen work, together with a brief discussion of the applicability of the standard method for the direct determination of nitrogen as free ammonia, in each instance showing by comparative data the relation between results by direct and distilled methods.

THE DIRECT KJELDAHL PROCESS.

In developing the direct Kjeldahl procedure for the soft-water sewage of Waterbury, attention was directed to the question of the interference of calcium and magnesium, which Columbus experience indicated to be the controlling feature in successful direct nesslerization. Analysis showed, however, that neither calcium nor magnesium was present in the Waterbury sewage to such an extent as to cause turbidity in a nesslerized tube.

After eliminating from the discussion the calcium and magnesium factors, the next step was to determine the effect of different grades

¹ *Jour. Infect. Dis.*, Supplm. 2, 1906, p. 109.

of caustic soda upon the color produced by Nessler's reagent; for, as the Columbus results indicated, the purity of the caustic soda used in neutralizing the acid digestates controls in a large measure the clearness of ammonia tubes when nesslerized.

Caustic soda from sodium was tried first and with highly satisfactory results, but the cost seemed too high for routine laboratory work. Caustic soda by alcohol was next used. No cloudiness resulted, but, with increasing quantities of the alkali, the depth of the color produced increased slightly, as has been found by former experience.^{1, 2}

The grade of caustic soda commonly known as "Purified Sticks" did not give satisfactory results. When fully purified the "blank" was high and the amount of preparatory treatment necessary was not commensurate with the difference in cost between this product and that by alcohol.

THE WATERBURY METHOD.

Having satisfactorily disposed of the question of turbidity and that of the most suitable grade of caustic soda, our attention was directed so to modify details of the method used at Columbus as to develop a process which would be practical and reliable under Waterbury conditions. After some experimentation the method finally assumed the following definite form.

Method.—A suitable portion of the sample (50 to 100 c.c.) is placed in a 300 c.c. Kjeldahl flask, diluted to a volume of 150 c.c. and boiled down to 25 c.c. to insure the removal of the nitrogen as free ammonia. The usual amount of sulphuric acid is then added, and the digestion is then carried to completion. After cooling, the contents of the flask are removed into a 100 c.c. graduated flask, thoroughly shaken, and from the sample there are removed 50 c.c. and placed into a second 100 c.c. flask. The caustic soda is now added, just short of the neutral point; the flask is cooled and the digestate made alkaline, adding in excess not more than 1 c.c. of a 25 per cent caustic-soda solution. The solution is then poured into a dry four-ounce bottle which is tightly stoppered, and set aside for twenty-four hours. During this period it is advisable to agitate the bottle slightly, so as to detach any suspended particles that may cling to the sides.

At the end of 24 hours, or at least of 12 hours, 10 c.c. of the clear supernatant liquor are then removed and placed into a 50 c.c. Nessler tube to which there has been previously added about 25 c.c. of ammonia-free water.

The tubes are next filled to the mark and are mixed by inverting three or four times; the tubes are then nesslerized and read in the usual manner.

¹ NESSLER, *Tiemann-Gartner's Handbuch*, 1905, p. 114.

² HAZEN AND CLARK, *Amer. Chem. Jour.*, 1890, 12, p. 425.

To compute the results, which, of course, are in terms of organic nitrogen, it is convenient to use the formula worked out by Kimberly and Roberts:¹

$$\frac{(N \times 20) - \text{Blank} \times 1,000,000 \times .00001}{S};$$

where N = c.c. standard reading, S = c.c. sample digested.

DISCUSSION OF DETAILS OF METHOD.

In the Columbus method no provision was made for first removing the free ammonia before the digestion. In applying the Columbus method to the work at Waterbury, it was not found practicable to obtain results comparable with those of the distillation process, nor with each other, for the probable reason that the quantity of sample taken for analysis, especially in the case of strong sewages, was so small as to introduce a large factor of error. To avoid this, it was decided to remove by boiling the nitrogen as free ammonia, thus permitting the use of a greater quantity of the sample. In the opinion of the writer, the necessity for the removal of the nitrogen as free ammonia before digestion, is probably controlled in a large measure by the relative amounts of nitrogen in that form and as organic nitrogen, although he favors the complete removal of the nitrogen as free ammonia.

It was found important to measure accurately the quantity of caustic soda to be used in neutralizing the acid digestates. For this purpose it was found convenient to arrange a four-liter bottle of the caustic soda in such a position that the solution could be blown into a burette whereby the amount of alkali could be accurately measured.

The necessity for using in excess not more than 1 c.c. of a 25 per cent caustic-soda solution lies in the fact that tubes which contain a large excess of alkali do not mix well, on account of the difference between the specific gravity of the dilution water and that of the alkali; and further because samples which are barely alkaline never become entirely free from suspended particles. According to the writer, a failure to observe either one of the precautions is sure to result in cloudy tubes.

COMPARISON OF RESULTS BY DIRECT AND DISTILLED METHODS.

To judge of the feasibility of adopting the direct process, as a routine procedure, a large number of analyses were made, comparing

¹ *Jour Infect. Dis.*, Supplm. 2, 1906, p. 117.

the results of the direct process with those of the distilled, as obtained according to the standard procedure. Representative analyses are listed below, and a study of the results indicates that the direct method is capable of affording very satisfactory results, which agree closely with the results of the distillation method.

TABLE I.
EFFICIENCY OF DIRECT PROCESS FOR DETERMINING KJELDAHL NITROGEN AS COMPARED WITH THE DISTILLATION METHOD.

SOURCE OF SAMPLE	ORGANIC	
	Direct	Distilled
Crude sewage.....	7.0 7.3 7.0	7.0 7.4 6.9
Septic sewage	18.0 16.4 11.5 15.0 10.7 8.7	19.0 16.3 12.2 14.8 11.2 8.4
Sprinkling filter effluent	12.0 10.5 9.8	12.0 11.0 9.5
Effluent contact filter	7.8 5.2 9.0	8.0 5.0 9.4
Sand filter effluent.....		

APPLICATION OF THE DIRECT KJELDAHL PROCESS TO THE DETERMINATION OF NITROGEN IN SEWAGE SLUDGE.

Following the general scheme outlined for the analysis of samples of sewage and effluent, the direct Kjeldahl process was applied to sludge analysis. The procedure adopted was as follows:

An amount of sample is taken that will yield from 0.005 to 0.02 gm. of nitrogen; usually about 0.5 gm. of dried sludge lies within the above limits. Twenty c.c. of sulphuric acid are then added and the digestion continued until the digestate becomes clear. After cooling, the contents of the flask are rinsed into a 250 c.c. graduated flask, and made up to the mark with ammonia-free water. Twenty-five c.c. of this solution are next introduced into a 200 c.c. flask made slightly alkaline with caustic soda, exercising precautions similar to those necessary in sewage work, the solution made up to 200 c.c. poured into an eight-ounce bottle and allowed to stand for 24 hours. Five c.c. of the clear supernatant liquid are then removed and nesslerized in the usual way.

Very satisfactory results have been obtained by this method, as indicated by the table below in which there are presented the results of duplicate analysis of sewage sludge.

TABLE 2.
REPRESENTATIVE DUPLICATE RESULTS OF NITROGEN DETERMINATION IN SEWAGE SLUDGE.

SOURCE OF ANALYSES	DUPLICATE ANALYSES	
	Per Cent Nitrogen	Per Cent Nitrogen
Grit chamber sludge	4.74 1.66 1.15 2.37 2.04 1.98 1.60 1.60 2.00	4.50 1.24 1.34 2.40 1.85 2.10 1.60 1.60 1.97
Settling tank sludge.....		
Septic sludge.....		

NOTES ON THE DIRECT DETERMINATION OF FREE AMMONIA BY THE STANDARD METHOD.

In determining nitrogen as free ammonia by a direct process, the method outlined by the Committee on Standard Methods of the Public Health Association has been followed, except that the sewage is filtered before the addition of the caustic soda and the copper sulphate.^{1, 2}

Necessity for filtration.—The idea of a preliminary filtration of the sample was suggested by the fact that when the unfiltered sewage was treated there remained in suspension from 30 to 50 parts of colloidal matter, as against from 10 to 15 parts, in case a preliminary filtration was included in the technique.

From experience gained during the work at Waterbury it has been found that in case the supernatant liquid, from a direct nitrogen as free ammonia determination, contains more than 30 parts per million of colloidal matter; high results will be obtained even though but 5 c.c. of the supernatant are removed and then diluted 10 times. As above mentioned, this feature is entirely eliminated by passing the sample through filter paper before beginning the determination.

Entrainment.—In all direct processes there is of course afforded an opportunity for slightly low results, owing to the entrainment of ammonium salts by the forming of precipitates. In agreement with the results obtained by other investigators,³ the Waterbury results

¹ FARENSTEINER, BUTTENBURG, AND KORN, *Leitfaden f. d. chem. Untersuch. von Abwasser*, Hamburg, 1902.

² PHELPS, "Determination of Ammonia in Sewage," *Jour. Infect. Dis.*, 1904, 1, p. 327.

³ KIMBERLY AND ROBERTS, *Jour. Infect. Dis.*, Supplm. 2, 1906, p. 119.

indicate that the nitrogen lost by this means may be considered a negative quantity. This, in the opinion of the writer, may be inferred from the table comparing the results of Kjeldahl nitrogen determinations by direct and distilled processes.

As evidence to support the view that the entrainment factor is by no means of practicable moment, there is presented below a table showing comparative results of nitrogen as free ammonia determined by the direct and distillation processes respectively.

TABLE 3.
RESULTS OF NITROGEN AS FREE AMMONIA BY DIRECT AND BY THE DISTILLATION PROCESSES

Source of Sample	Direct	Distilled
Crude sewage.....	17.5	18.0
	10.3	10.3
	17.0	16.8
Septic sewage.....	12.0	12.0
	9.5	9.4
	9.8	8.9
Sprinkling filter effluent.....	6.2	5.8
	6.1	6.1
	5.0	5.6
Sand filter effluent.....	2.5	2.0
	3.8	4.5
	2.4	2.4

CONCLUSIONS.

In conclusion it may be said that the direct process for the determination of Kjeldahl nitrogen, and that for the determination of nitrogen as free ammonia, have been entirely satisfactory under Waterbury conditions. Out of some 4,000 or 5,000 analyses, not more than 10 per cent of the determinations have been in error, due to cloudy nesslerized tubes, and in almost every case the cause of turbidity could be traced to some error of technique and not to any inherent fault in the process itself. Furthermore, by adopting the above methods it has been possible to increase the number of samples analyzed, and to obtain just as efficient results as if the distillation method had been used.

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